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## Study on grinding of black pepper and effect of low feed temperature on product quality

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### Abstract

The effect of feed temperature on grinding of black pepper (*Piper nigrum*) in a hammer mill was studied at Coimbatore (Tamil Nadu). Variation in temperature was attained by keeping black pepper in a refrigerator maintained at 4°C (low temperature sample) to attain a feed temperature of 7.9 to 21.2°C and by mixing with dry ice at different ratios (ultra low temperature sample) to get temperatures from -3.33 to 12.86°C. On the basis of volatile oil content, oleoresin, piperine and volatile components, a feed temperature of -3.33 to -1.25°C was optimized. The volatile oil content of the product indicated an increase of about 17% retention of oil for ultra low temperature and 15% for low temperature compared to the ambient condition (control). Gas chromatographic analysis of the oil showed higher retention of monoterpenes under ultra low temperature condition compared to ambient and low temperature conditions of grinding. An increase of 1% to 3% moisture was observed for the ultra low temperature sample for a feed temperature of -3.33 to -1.25°C, which helped to increase 2% to 3% oleoresin yield. The volume surface mean diameter of black pepper powder showed an increasing trend of 0.15 mm to 0.18 mm with an increase in feed temperature from -3.33 to 12.86°C for ultra low temperature sample. The specific energy consumption varied from 24.79 to 46.62 kJ(kg°C)<sup>-1</sup> with increase in grinding temperature.

**Keywords:** black pepper, grinding temperature, *Piper nigrum*, quality.

### Introduction

Black pepper (*Piper nigrum* L.) contains about 3.8% volatile oil and the major constituents of the oil are low boiling monoterpenes, high boiling sesquiterpenes and oxygenated compounds (Sankarikutty *et al.* 1982). Grinding is an important step in post harvest

operation, requiring special attention as it involves the additional problems of volatility and loss of aroma. Grinding facilitates the release of aroma/flavour principles and better uniform mixing with food materials. At the same time, grinding of spices results in considerable loss of aroma due to the heat

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generated (42 to 95°C) during conventional grinding (Gopalakrishnan *et al.* 1991). The loss of volatile oil can be considerably reduced by cryogenic grinding technique (Pruthi 1980). Grinding of spices under cryogenic conditions causes distortion in the natural composition. Pre-cooling of the raw spice and the continuous low temperature maintained within the mill reduces loss of volatile oil, thereby, retaining most of the flavour strength per unit mass of spice. The present paper reports the effect of grinding temperature on specific energy consumption, volume surface mean diameter of black pepper powder, and the retention of the quality of black pepper in the temperature range of -3.33°C to 40°C.

### Materials and methods

The study was conducted at the College of Agricultural Engineering, Tamil Nadu Agricultural University, Coimbatore. Black pepper procured from a local trade centre was used for the study. The volatile oil content, initial moisture, oleoresin and piperine contents were estimated as per AOAC (1975) procedures. Gas chromatographic analysis of the oil was carried out for the major constituents namely, pinene, myrcene, limonene and  $\beta$ -caryophyllene.

A hammer mill was used for grinding the samples. Five thermocouples, along with a digital temperature indicator of  $\pm 0.01^\circ\text{C}$  accuracy, were used for measuring the temperature. In order to record the correct temperature in the grinding zone, three thermocouples were placed in the sieve at different locations; one at the centre and the other two at the sides at equal distance. The fourth thermocouple was placed in the feed hopper and the last one at the collecting pan for measuring the product temperature.

The ground sample was packed in moisture resistant flexible pouches and stored in a refrigerator at  $4^\circ\text{C}$  till they were analysed for average particle size, volatile oil content, gas chromatogram of oil, piperine content, and oleoresin yield.

Four different sieve sizes namely, 0.5 mm, 1.0 mm, 1.5 mm and 2.0 mm were used at the discharge end to study the effect of particle size on quality of black pepper powder. The grinding was performed by impact and attrition. The sieve at the discharge end controls the final product size. The method of sieve analysis, employing vibratory type sieve shaker was used for the determination of particle size. The fractions retained on all the sieves after 20 min of shaking were weighed using a physical balance having an accuracy of  $\pm 0.001\text{g}$ . The volume surface mean diameter which gives an overall representation of the size of the ground sample was calculated using the equation:

$$D_{vs} = \frac{1/4}{\sum (Q_i/D_i)}$$

where,  $D_{vs}$  = volume surface mean diameter in mm;  $Q_i$  = weight fraction of total sample retained by  $i^{\text{th}}$  screen;  $D_i$  = average opening of the  $i^{\text{th}}$  screen in mm.

The grinding experiments were carried out under three different temperature conditions namely, ambient condition (30 to  $40^\circ\text{C}$ ), low temperature condition (grinding the sample maintained at  $4^\circ\text{C}$  in a refrigerator for 6, 8, 10, 12 and 14 days) and ultra low temperature condition (grinding of sample along with dry ice in different ratios namely, 2:1, 4:1, 6:1, 8:1 and 10:1) to get feed temperatures of -3.33, -1.25, 2.34, 8.38 and  $12.86^\circ\text{C}$ . The sample taken for each trial was 500 g. Each experiment was replicated three times.

The time taken for each grinding experiment was noted with a stopwatch. For the measurement of energy, the grinder was connected to a three phase 15 ampere energy meter. Specific energy consumption was obtained by dividing the energy requirement during grinding by the feed rate. The quality of the powder was assessed by measuring the percentages of volatile oil, oleoresin and piperine content and by gas chromatographic analysis of volatile components.

## Results and discussion

The volatile oil content was maximum for samples sieved through 2 mm size sieve. For getting the particle size, which satisfies the Indian standard, 1 mm sieve size was selected and used for further studies. The decrease in volatile oil content in the case of 1 mm sieve when compared to 2 mm sieve could be reduced by controlling the rise in temperature at the grinding zone. Volatile oil content as a function of sieve size is presented in Table 1.

**Table 1.** Variation in volatile oil percentage in black pepper with sieve size

Sieve size (mm)	Volatile oil (%)
1.0	2.18
1.5	2.26
2.0	2.33

It was observed that during the grinding experiment at ambient conditions, the temperature at the grinding zone increased from 40.30 to 47.45°C in 40 min. While grinding the ultra low temperature sample, it was observed that, for a feed temperature of -3.33°C (which was the minimum), the grinding zone temperature reached 23.13°C. For a feed temperature of 21.2°C (which was the maximum) for low temperature sample, a grinding zone temperature of 39.95°C was observed. This increase in temperature was due to the heat generated during grinding (Pruthi & Misra 1963).

The volume surface mean diameter decreased from 0.18 mm to 0.16 mm with increase in feed temperature from 7.9 to 21.2°C in the case of low temperature grinding of black pepper. But in the case of ultra low temperature sample, the volume surface mean diameter showed an increasing trend of 0.15 mm to 0.16 mm with increase in feed temperature from -3.33°C to 12.86°C. This may be attributed to the fact that at higher temperature, brittleness of the product decreased resulting in coarser grinding of the powder. Similar characteristics of the particle size distribution of ground caraway powder

at different grinding temperatures (0 to 44°C) were reported by Wolf & Pahl (1990). The variation in volume surface mean diameter with the feed temperature in the case of low temperature grinding conditions and ultra low temperature grinding conditions can be represented as:

For low temperature grinding sample

$$D = 0.2002 - 0.002 T \quad (r^2 = -0.896) \quad (1)$$

For ultra low temperature sample

$$D = 0.1516 + 0.0006 T \quad (r^2 = -0.986) \quad (2)$$

Where, D=Volume surface mean diameter in mm, and T=Feed temperature in °C.

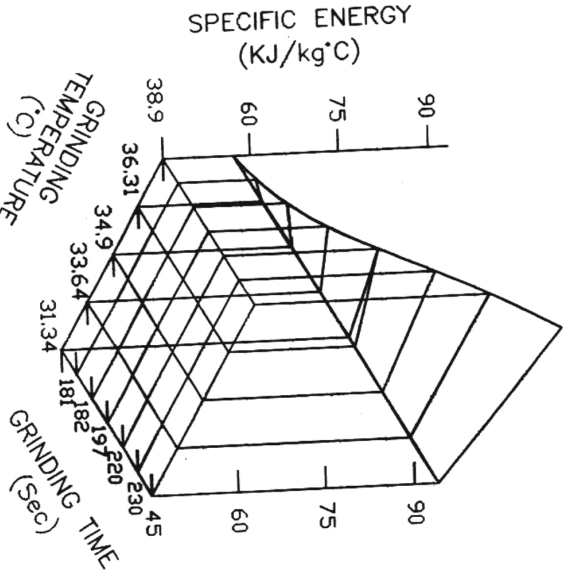
There was an increase in grinding time with decrease in feed temperature. At feed temperature of 21.2°C the time taken for grinding was 181 sec, whereas at 7.9°C it was 230 sec. The trend may be due to the absorption of moisture by black pepper during storage in the refrigerator for 14 days. A similar trend was observed for cardamom by Gopalakrishnan *et al.* (1991). When the grinding time increased, specific energy required for grinding black pepper also increased. In the case of ultra low temperature sample, as the feed temperature increased from -3.33°C to 12.86°C, the grinding time increased from 151 to 233 sec (Figs. 1 and 2). It is obvious that at low feed temperature, the degree of brittleness of black pepper increased, which resulted in less grinding time. Similar results were obtained by Murthy *et al.* (1996).

Increasing the grinding zone temperature resulted in reduction in volatile oil, from 2.32% to 2.11% for a grinding zone temperature of 31.34 to 39.99°C for low temperature sample. Similarly, in the case of ultra low temperature sample, reduction of 2.36% to 2.28% of volatile oil was noticed for a temperature rise of 23.13 to 40.06°C (Table 2). Statistical analysis of the data revealed that the variation in volatile oil content was significant ( $P=0.01$ ) for all the grinding

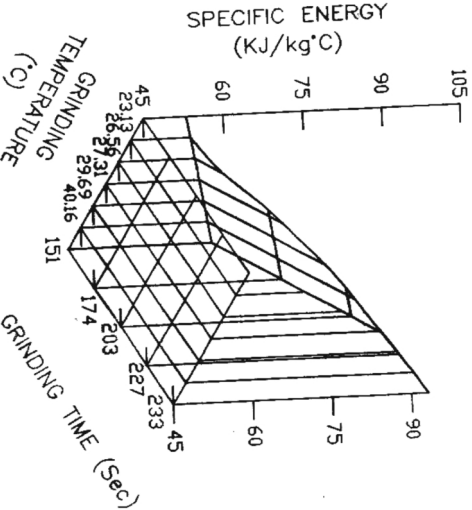
**Table 2.** Volatile oil content of black pepper powder ground under different temperature conditions

Component	Ambient sample			Low temperature sample					Ultra low temperature sample				
Feed temp. (°C)	30.0	35.0	40.0	7.9	11.7	15.3	17.5	21.2	-3.33	-1.25	2.34	8.38	12.86
Grinding zone temp. (°C)	40.3	42.97	47.45	31.34	33.64	34.90	36.31	39.99	23.13	26.56	26.31	29.69	40.06
Volatile oil (%)	2.26	2.16	2.02	2.32	2.23	2.20	2.20	2.11	2.36	2.34	2.30	2.30	2.28
	(±0.02)	(±0.03)	(±0.01)	(±0.02)	(±0.01)	(±0.01)	(±0.01)	(±0.01)	(±0.01)	(±0.01)	(±0.01)	(±0.01)	(±0.02)
Oleoresin (%)	10.25	10.09	9.31	13.15	12.25	11.20	10.75	10.33	14.95	14.79	13.95	13.95	13.80
	(±0.09)	(±0.01)	(±0.05)	(±0.04)	(±0.04)	(±0.04)	(±0.04)	(±0.04)	(±0.05)	(±0.008)	(±0.05)	(±0.04)	(±0.04)
Piperine (%)	5.28	5.1	5.76	6.25	6.15	5.5	5.48	5.4	6.32	6.28	6.25	5.93	5.81
	(±0.02)	(±0.02)	(±0.03)	(±0.04)	(±0.04)	(±0.04)	(±0.02)	(±0.04)	(±0.02)	(±0.02)	(±0.08)	(±0.05)	(±0.08)

Values in parenthesis are standard deviation



**Fig. 1.** Effect of grinding zone temperature on grinding time and specific energy (Low temperature sample)



**Fig. 2.** Effect of grinding zone temperature on grinding time and specific energy (Ultra low temperature sample)

conditions. During the grinding process the mass transfer increased because of increase in vapour pressure at higher temperatures, which resulted in the loss of volatile oil (Wolf & Pahl 1990). Similar trend of reduction in volatile oil with increase in grinding zone temperature from 10 to 50°C for black pepper samples was also reported by Landwehr & Pahl (1986) and it might be owing to the fact that the black pepper oil consist of low boiling components which evaporated at this corresponding temperature. About 15% more retention of volatile oil was observed in the case of low temperature sample and 17% for ultra low temperature sample compared to ambient condition sample (2.02 ml). This result is supported by of Murthy *et al.* (1996)

There was a better retention of monoterpenes (pinene, myrcene and limonene) for low temperature and ultra low temperature samples compared to ambient condition sample (Table 3). The analysis of variance indicated that the variation of the monoterpenes with grinding zone temperature was significant ( $P=0.01$ ). The variation in the value of sesquiterpenes was due to the incomplete distillation of volatile oil. Increase in grinding zone temperature resulted in reduction in oleoresin content (Table 2). This may be due to loss in volatile oil at high temperature. In the case of ultra low temperature sample, the retention of oleoresin was more when compared to low temperature and ambient samples. This may be due to the retention of constituents of oleoresin along with volatile oil and piperine. Similar results were also obtained for nutmeg (Mckee *et al.* 1993). In the case of ultra low temperature sample, for a feed temperature of -3.33°C and -1.25°C, 2% to 3% increase in oleoresin content was observed compared to the test sample content. During grinding along with dry ice, the product might have absorbed moisture which resulted in increase yield in oleoresin.

Piperine content as a function of grinding zone temperature showed a decreasing trend with increase in grinding zone temperature

**Table 3.** Volatile components of volatile oil of black pepper powder ground under different temperature conditions

Component	Ambient sample			Low temperature sample			Ultra low temperature sample						
Grinding zone temp. (°C)	40.3	42.97	47.45	31.34	33.64	34.90	36.31	39.99	23.13	26.56	26.31	29.69	40.06
Pinene	2.314	1.223	0.831	4.123	3.591	3.153	2.812	2.681	6.586	6.238	5.001	3.883	3.921
	(±0.001)	(0.001)	(±0.02)	(±0.002)	(±0.002)	(±0.003)	(±0.002)	(±0.001)	(±0.04)	(±0.01)	(±0.005)	(±0.01)	(±0.003)
Myrcene	9.461	9.107	8.281	13.033	12.574	12.129	11.845	10.533	14.635	13.990	12.115	12.021	10.481
	(±0.02)	(±0.001)	(±0.001)	(±0.001)	(±0.002)	(±0.01)	(±0.003)	(±0.02)	(±0.02)	(±0.004)	(±0.002)	(±0.003)	(±0.02)
Limonene	9.108	8.820	8.568	11.022	10.964	10.785	9.644	8.847	12.715	12.473	12.3.9	12.109	10.372
	(±0.001)	(±0.002)	(±0.003)	(±0.002)	(±0.003)	(±0.002)	(±0.002)	(±0.001)	(±0.02)	(±0.01)	(±0.01)	(±0.01)	(±0.01)
b-Caryophyllene	19.118	18.506	16.713	30.170	26.782	28.833	28.953	28.656	30.084	26.087	28.0320	28.332	26.914
	(±0.001)	(±0.01)	(±0.003)	(±0.03)	(±0.001)	(±0.001)	(±0.002)	(±0.002)	(±0.03)	(±0.04)	(±0.02)	(±0.01)	(±0.02)

Values in parenthesis are standard deviation



for all samples (Table 2). It was observed that variation in temperature had no influence over piperine content for the ambient samples whereas the low temperature and ultra low temperature samples had highly significant influence ( $P=0.01$ ). The ultra low temperature sample was observed to have a higher piperine content than the low temperature sample for all the grinding zone temperature levels.

The moisture content of black pepper powder immediately after grinding was analyzed. It was observed that in the case of ambient and low temperature samples there was no increase in moisture content but in the case of ultra low temperature sample (with 2:1 and 4:1 ratio), there was an increase in moisture by 3% and 2%, respectively. It might be due to the absorption of moisture by black pepper during grinding along with dry ice. Similar results were observed by Gopalakrishnan *et al.* (1991) in cardamom.

The study indicated that volume surface mean diameter of ground black pepper powder increased at lower temperature. There was increase in grinding time with decrease in feed temperature for refrigerated samples. Increase in grinding time and specific energy for increased feed temperature was noted in dry ice mixed sample. Volatile oil decreased with increase in grinding temperature; volatile oil retention was higher by 15% and 17% in refrigerated and dry ice mixed sample respectively, compared to ambient sample. Constituents like pinene, myrcene and limonene and oleoresin and piperine decreased with increase in grinding temperature. An increase of 27% to 30% in oleoresin was noted in dry ice mixed sample at 2:1 and 4:1 ratios. The study would help in value addition of black pepper.

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